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Designer Nanocellular Materials for Laser Targets and Other DNT Applications

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Overview. This document and the accompanying manuscripts summarize the technical accomplishments of our one-year LDRD-ER effort, a project that has since been incorporated into a larger LDRD-SI for FY05. The objective of this effort was to develop a predictive synthetic capability for the preparation of materials with cellular architectures (sub-micron pore or cell sizes and relative densities less than 10% of full density) not attainable by conventional methods. The ability to reliably prepare these nanocellular materials and control their bulk physical properties (e.g. mechanical strength) would be a considerable advance in the areas of porous materials and its impact would cut across many existing LLNL investments. One significant area related to the Laboratory mission that would benefit is the design of new materials for high energy density physics (HEDP) targets. Current synthetic techniques do not allow for the preparation of foams that meet all of the current and projected compositional and mechanical requirements of these experiments. This project focused on two main types of materials: inorganic sol-gel materials and nanocellular metal foams. The following sections describe the project goals for these two types of materials as well as the progress made towards these goals in FY04. These sections also provide context for the three publications that have been included in this final report.

Structure Control in Sol-Gel Materials. The sol-gel process involves the transformation of reactive monomers into nanometer-sized clusters that crosslink to form a three-dimensional gel network. Due to the colloidal nature of the process, sol-gel materials typically possess sub-micron architectures that are defined by randomly interconnected particles with diameters in the 1-100 nm range. Due to their unique microstructure, materials prepared by the sol-gel process are being investigated for a wide range of applications including catalysis, separation technology, ceramics, sensors/detectors and nano-electronics. One of the shortcomings of current sol-gel chemistry, however, is the inability to reliably control and predict the bulk physical properties of these materials. The final properties of the sol-gel material are closely associated with several factors, such as precursors, catalyst and reaction conditions. These synthetic variables can affect the kinetics of nucleation, particle growth and the organization of the gel network. These parameters, in turn, have a pronounced influence on the macroscopic properties (e.g. mechanical strength, surface area, porosity) of the sol-gel products. Control over bulk physical properties, therefore, requires a fundamental understanding of the mechanisms by which solution species form, aggregate, and assemble into the gel network, and how these processes influence the physical properties of the sol-gel materials. The development of such relationships has not yet been fully realized for these materials.

In order to obtain this detailed insight, an *in-process* characterization technique is required that can probe the structure and composition of the solution species as well as monitor their organization into the final gel microstructure. Nuclear magnetic resonance spectroscopy (NMR) is ideally suited for this level of characterization since the technique provides characteristic signatures that can track changes in both solid and solution phases in an elementally selective and non-destructive manner. Our goal in this effort is to establish a correlation between

synthetic variables and material properties analogous to those used to develop structure-property relationships in organic polymer systems. We are using solution, gel and solid-state NMR techniques to follow the transformations associated with the sol-gel process and the information derived from these experiments should provide detailed insight into the relationship between synthesis and the macroscopic properties in inorganic sol-gel materials. Such an advance would fundamentally change the way these materials are prepared and, as a result, expand the technological potential of sol-gel science. As a first step in this effort, we are applying these NMR techniques to monitor structure formation in aluminum oxide (Al_2O_3) aerogels. For FY04, our goals were to: (1) prepare Al_2O_3 aerogels from formulations that produce at least two distinct structural morphologies, (2) use solution NMR techniques to follow reactions that occur during sol formation and (3) use solid-state NMR techniques to characterize the microstructure of the final sol-gel material. Since the start of this effort, we have developed an Al_2O_3 aerogel system that, by changing a single synthetic variable, generates a set of materials whose morphology and mechanical properties are dramatically different. We have also characterized these Al_2O_3 systems using both solution and solid-state NMR techniques. The bulk of this work is described in the attached manuscript entitled "Synthesis of High Surface Area Alumina Aerogels without the Use of Alkoxide Precursors".

Nanocellular Metal Foams. Commercial metal foams are synthesized by a wide variety of methods and typically have relative density in the range of 0.03 to 0.3, and cell sizes in the range of 2 to 10 mm. Current foaming techniques, however, are inadequate for synthesizing metal foams with nanocellular architecture. The goal of this project is to establish a new foaming technology for synthesizing novel micro- or nanocellular metal and composite foams. We are utilizing a method known as dealloying to control the cell sizes in porous metals. This approach has been used to synthesize novel foams from high Z metals such as Au and Pt. The dealloying method involves a phase separation process (spinodal decomposition) within bulk Au-Ag, Au-Cu, and Pt-Cu alloys through heat treatment at elevated temperatures. Nanoporous Au or Pt sponges (open-cell foam) can then be produced by selectively leaching out the less noble component of the alloy, i.e. Ag from Au-Ag alloy or Cu from Pt-Cu alloy, using an electrochemical process. An additional goal of this effort is to advance knowledge of materials science as a result of the investigation of surface-controlled foaming mechanisms and surface-controlled deformation mechanisms and kinetics within the novel foams under quasi-static and dynamic pressure conditions. The grain structure, cellular architecture and mechanical properties (modulus, strength, creep strength, strain-rate sensitivity and dynamic behavior) of these nanocellular metal foams will be characterized by a variety of techniques. Emphasis is being placed on correlating measured properties with structural parameters and mechanical properties of the foams. For FY04, we proposed to initiate de-alloying efforts to: (1) reproduce nanocellular Au foam work (relative density > 20%), (2) characterize architecture, microstructure, and mechanical properties of nanocellular Au. Since the start of this effort, we have prepared dealloyed Au foams at 40% and 30% relative density and characterized the structural and mechanical properties of these materials. These efforts are described in the attached manuscripts entitled "Nanoporous Au: A high yield strength material" and "Monolithic Nanocrystalline Au Fabricated by the Compaction of Nanoscale Foam".

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